

# One step coating of Ni – Fe alloy outerwear around 1-3 dimensional nano materials by novel technology

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## The detailed of electrodeposition

An electrochemical cell with a two-electrode configuration was used for the experiments. A copper plate (30 mm×30 mm×1 mm) was used as the anode and another copper plate (10mm×10mm×1mm) used as cathode.

## Nickel iron plating formula

Nickel (II) sulfate hexahydrate 100 g/l  
Iron (II) sulfate heptahydrate 10 g/l  
Boric acid 40 g/l  
Sodium chloride 25 g/l  
Saccharin 2 g/l  
Sodium citrate 14.7 g/l  
Ascorbic acid 0.5 g/l  
2-Butyne-1,4-diol 0.6 g/l  
Sodium dodecylbenzenesulphonate 0.05 g/l

**Table S1** Plating conditions

Temperature	60 °C
pH	4.2
Plating time	600 s
Current density	6 A/dm <sup>2</sup>

## Characterization

The morphologies of Ni-Fe alloy@1-3 dimensional core-shell nano materials were observed using a TECNAI G<sup>2</sup>Tf<sup>20</sup> transmission electron microscope, TEM (FEI American). All samples for TEM characterization were prepared on copper wire mesh. The X-ray diffraction (XRD) measurements were performed on a Rigaku D/max-2400 diffractometer, using Cu-K $\alpha$  radiation as the X-ray source in the range of 20–80°. Hysteresis loop measurement was used to characterize the magnetism of Ni-Fe alloy@1-3 dimensional core-shell nano materias. The content of Ni-Fe alloy@1-3 dimensional core-shell nano materials was analyzed by inductively coupled plasma (ICP) spectroscopy using an IRIS Advantage analyzer. X-ray photoelectron spectroscopy (XPS) was recorded on using an Kratos AXIS Ultra DLD instruments with a monochromatic X-ray source (Al K $\alpha$   $h\nu = 1486.6$  eV) and all the peaks were calibrated by the standard position of C 1s peak.

## Preparation of working electrode

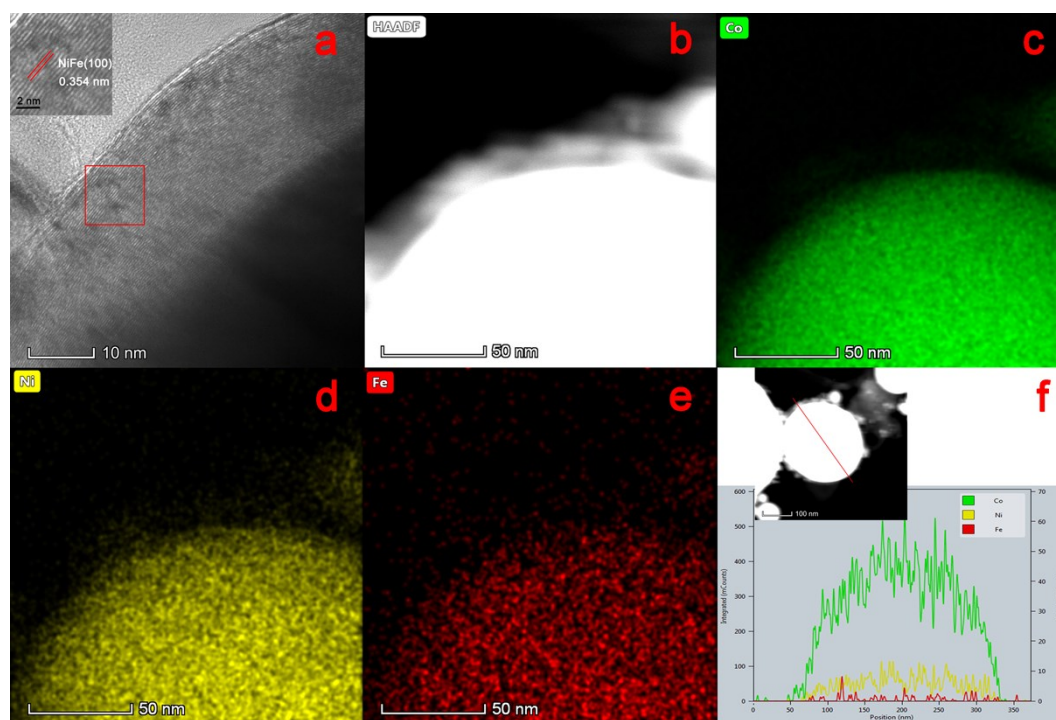
The glassy carbon electrode (GCE, 3.0 mm in diameter, Wuhan Gaoss Union Technology Co., Ltd.) was polished mechanically with Al<sub>2</sub>O<sub>3</sub> powder (Aldrich, 0.05  $\mu$ m) and then cleaned with ethanol and deionized water. The catalyst ink was formed with 1 mg catalysts dispersed in 1 mL water-ethanol-Nafion (volume ratio = 2 : 1 : 1, Nafion : 0.5 wt%) solution, followed by sonication for at least 30 min. Then, 10  $\mu$ L of well-dispersed catalyst ink (containing 0.01 mg of catalyst) was loaded onto the GCE and dried 10 min under an infrared light to form the working electrode with a loading of 0.143 mg cm<sup>-2</sup>.

## C<sub>dl</sub> measurement

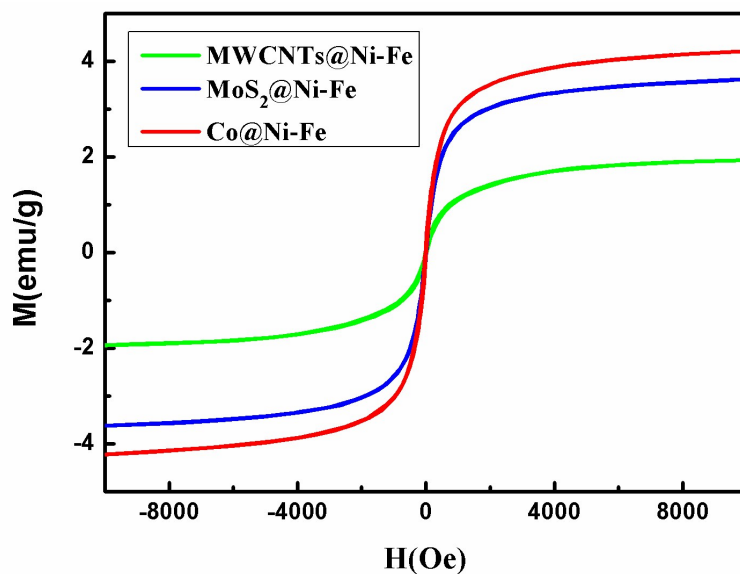
The double-layer capacitance (C<sub>dl</sub>) measurement of Ni-Fe alloy@1-3 dimensional core-shell nano materials was operated by cyclic voltammetry (CV) in 0.1 M KOH solution at room temperature with a typical three-electrode system as above. The cyclic voltammetry (CV) was performed in a non-faradaic region (-0.05–0.05 V vs. Hg/HgO) at different scan rates (2–10 mV s<sup>-1</sup>). The data of C<sub>dl</sub> was equated by the linear slope of the one-half of the difference in current density at 0 V vs. Hg/HgO plotted against scan rate. The current density was normalized to the geometrical surface area of the GCE (0.07 cm<sup>2</sup>).

**Table S2** The concentration of Nickel (II) and Iron (II) in plating solutions during polarization curve measurement

The concentration of Nickel (II) and Iron (II) in plating solutions				Mass percent
Nickel (II) sulfate	100 g/l	Iron (II) sulfate	10 g/l	100 wt%
Nickel (II) sulfate	90 g/l	Iron (II) sulfate	9 g/l	90 wt%
Nickel (II) sulfate	80 g/l	Iron (II) sulfate	8 g/l	80 wt%
Nickel (II) sulfate	70 g/l	Iron (II) sulfate	7 g/l	70 wt%
Nickel (II) sulfate	60 g/l	Iron (II) sulfate	6 g/l	60 wt%



**Figure S1** (a) HRTEM images of Ni-Fe alloy@Co NPs; (b) HAADF-STEM image of Ni-Fe alloy@Co NPs and the corresponding HAADF-STEM-EDX elemental mapping images of (c) Co, (d) Ni, and (e) Fe; (f) Cross-sectional compositional line profiles of Co, Ni and Fe in Ni-Fe alloy@Co NPs recorded along the line shown in the HAADF-STEM image (inset).



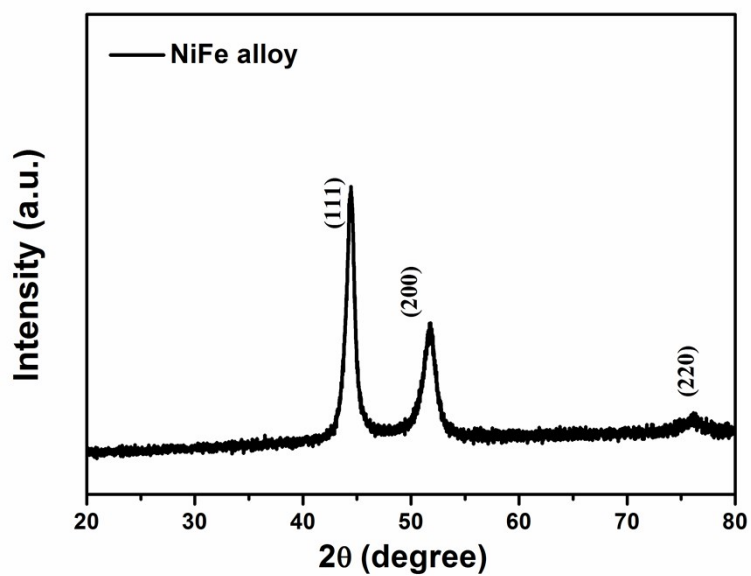
**Figure S2** Hysteresis loop curves of Ni-Fe alloy@MWCNTs, Ni-Fe alloy@graphene-like MoS<sub>2</sub>, and Ni-Fe alloy@Co NPs.

**Table S3** The compositions of the Ni-Fe alloy catalysts derived from inductively coupled plasma spectra (ICP)

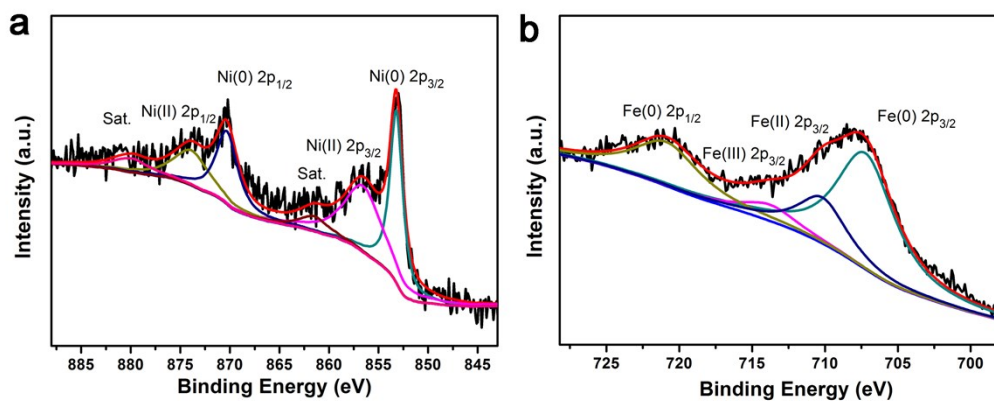
The concentration of Nickel (II) sulfate (g/L)	The quality of Iron (II) sulfate (g/L)	The compositions of the most active catalysts
100	2	Ni <sub>95.2</sub> Fe <sub>4.8</sub>
100	4	Ni <sub>92.9</sub> Fe <sub>7.1</sub>
100	6	Ni <sub>91.7</sub> Fe <sub>8.3</sub>
100	8	Ni <sub>90.2</sub> Fe <sub>9.8</sub>
100	10	Ni <sub>85.5</sub> Fe <sub>14.5</sub>
100	12	Ni <sub>80.1</sub> Fe <sub>19.9</sub>
100	14	Ni <sub>73.9</sub> Fe <sub>26.1</sub>
100	16	Ni <sub>71.1</sub> Fe <sub>28.9</sub>

**Table S4** The results of current efficiency measurement

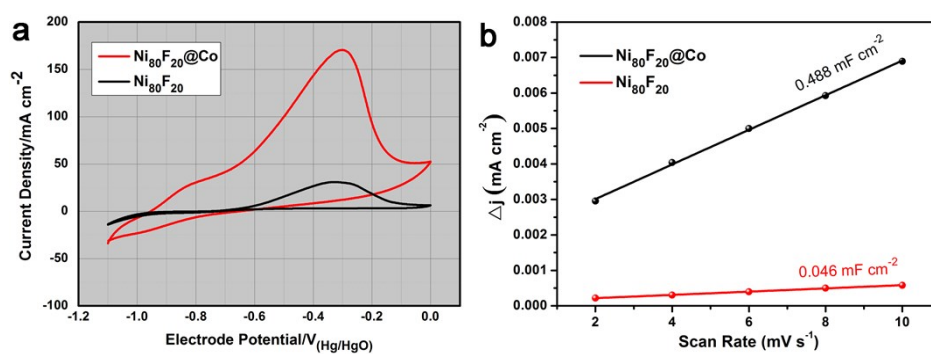
Electrodeposition time	The weight of Ni-Fe alloy (theoretical value) (mg)	The weight of Ni-Fe alloy (actual value) (mg)	Current efficiency
0-120s	4.39	1.39	31.66%
120-240s	4.39	1.21	27.56%
240-360s	4.39	1.13	25.74%
360-480s	4.39	1.08	24.60%
480-600s	4.39	0.95	21.64%



**Figure S3** XRD pattern of the reference Ni-Fe alloy catalyst.



**Figure S4** Deconvoluted high-resolution spectra of (a) Ni 2p and (b) Fe 2p of the reference Ni-Fe alloy catalyst.



**Figure S5** (a) Cyclic voltammetric measurements for hydrazine electrooxidation and (b)  $C_{dl}$  measurements of reference Ni-Fe alloy catalyst and  $Ni_{80}Fe_{20}@Co$  NPs.